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## A SIMPLIFICATION OF THE INVERSE-RATE METHOD FOR THERMAL ANALYSIS

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One of the most useful and at the same time least commonly used methods of thermal analysis for the determination of transformations in metals and alloys consists in the recording of the time intervals required for successive increments of temperature change during heating or cooling, the temperature of the furnace which contains the specimen being altered at a uniform rate. The curve obtained by plotting these time intervals as a function of the mean temperature of the specimen during the interval is called the inverse-rate curve. It is probably due to the fact that no simple and convenient method has apparently been available for the measurement of the successive time intervals that this method has not been so generally used as, for example, the differential method, for which, in addition, several types of automatic or semiautomatic apparatus have been designed.

Whenever this method has been used the intervals have usually been measured with the use of a chronograph. Its operation as practiced at the Bureau of Standards<sup>1</sup> is as follows: The temperature of the specimen is measured by thermocouple and dial potentiometer; the operator sets the potentiometer at successive values of the emf, differing by equal increments, usually 0.02 millivolt, and records the exact instant on the chronograph, by pressing a contact key, at which the galvanometer coil passes through its null position. Two-second intervals are also recorded

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<sup>1</sup> G. K. Burgess and J. J. Crowe, Critical Ranges, A<sub>2</sub> and A<sub>3</sub> of Pure Iron, this Bulletin, 10 (Scientific Paper No. 213); 1913.

on the same chronograph record, and the number of seconds elapsing between successive signals is afterwards counted from the record and plotted as a function of the emf, or of the temperature.

This method is an admirable one, and by it most minute thermal arrests may be detected, but it requires the use of a good chronograph, which is generally difficult to obtain, and the time of one operator during the recording and subsequently to read the intervals from the record. The latter operation often takes from one to two hours. There is here suggested a simple, convenient, and, it is believed, equally accurate method of recording the successive time intervals by which the expense of chronograph and the time of one operator in counting the chronograph record may be eliminated. The remainder of the apparatus, consisting of furnace, thermocouple, and potentiometer, is used exactly as in the former method.

Two stop watches are used, which may conveniently be mounted in a small frame to be held in one hand, a finger being placed on each stem. During the "run" the operator sets the potentiometer and marks the instant at which the galvanometer is at zero by pressing the stems of both watches simultaneously, stopping one at the end of the interval which it has measured and starting the other upon its measurement of the next. The interval is read and recorded upon a suitable blank sheet, the hand of this watch returned to its zero position, and the potentiometer set at the next value. The operation is repeated for each successive interval. The intervals so recorded are afterwards plotted directly as a function of the emf.

Fig. 1 shows the inverse-rate curves for two complete "runs," including the heating and the cooling curve, made on pure iron. Both  $A_3$  and  $A_2$  are indicated on the first set of curves, only  $A_2$  on the second set. In each case the intervals were recorded both with the stop watches and with the chronograph in the usual manner; the curves marked  $c$  were taken with chronograph, those marked  $w$  with stop watches. It is evident that there is little difference in the smoothness of the two sets of curves or in the accuracy or precision of measurement of the time interval.

A general consideration of the accuracy of the stop watch also indicates that the precision of measurement by stop watch is sufficient for the purposes of thermal analysis. It is only rarely, perhaps once in a hundred times, that a stop watch is not accu-

rate to within one-fifth of a second, and its maximum error is two-fifths of a second. It is found that the variation of successive intervals of time measured in the inverse-rate method, due to actual non-uniform rate of cooling or heating of the furnace, or to inaccuracy of the operator in signaling the moment when the potentiometer is balanced is of approximately the same value; that is, one-fifth of a second. It is therefore not necessary to

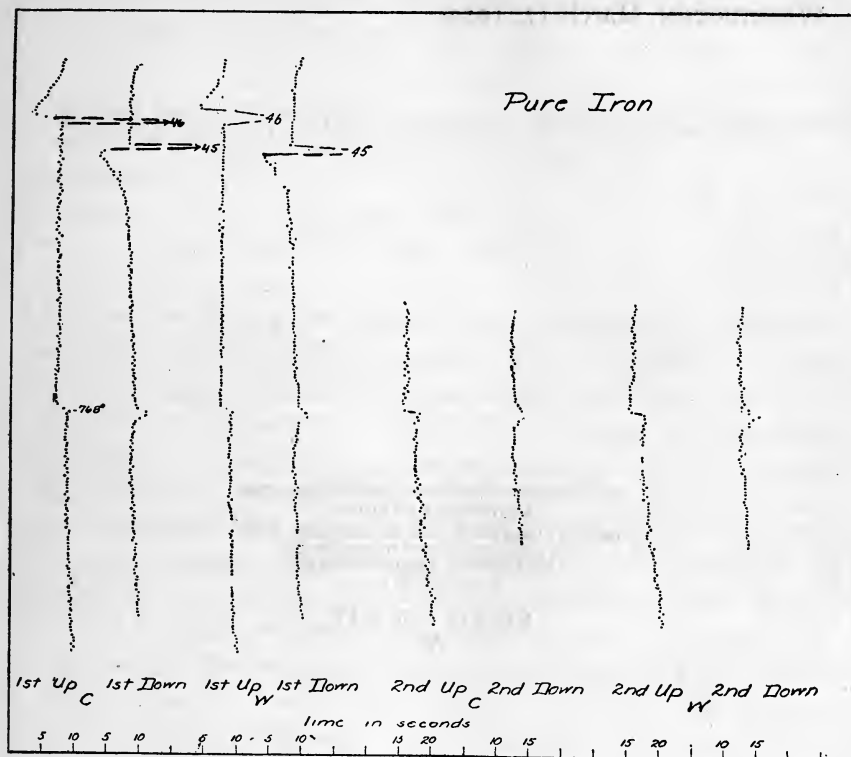


FIG. 1.—Heating and cooling inverse-rate curves of pure iron taken (C) with chronograph and (W) with stop-watch

obtain the greater precision of time measurement which is unquestionably possible by the use of the chronograph.

When the ordinary dial type of precision potentiometer is used, the operator has sufficient time for all of the operations necessary: setting the potentiometer, reading and recording the time interval from the watch, within an average interval of 15 seconds. This is recommended for usual conditions.

The curves recorded above were taken with a cheap variety of stop watch, costing about \$10. It does not appear that a more

expensive watch is necessary. It is, however, not advisable to use the type of stop watch which has also the usual hour and second hands, as the presence of so many indicating hands will only confuse the operator, who is obliged to read quickly and can not take much time in recognizing the stop hand.

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